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REPUBLIC
AVIATION CORPORATION

RAC 921-2
(RD-QPR-64-619-2)

ENGINEERING PROGRESS REPORT
ON INVESTIGATION OF THE BASIC
MECHANISM OF METAL FATIGUE

RAC 921-2

23 January 1964

Prepared under Naval Bureau of Weapons
Contract NOw 63-0704-c

Quarterly Report No. 2

1 October 1963 through 31 December 1963

This report applies to work
on contract: NOw 63-0704-c

REPUBLIC AVIATION CORPORATION
Farmingdale, L.I., N.Y.

ABSTRACT

Improved methods were devised for electrochemical cutting and chemical polishing of single crystal copper test specimens.

The mechanical loading system designed for the cyclic deformation of single crystals was calibrated and the reliability of strain measurements was determined.

Measurements were made of the attenuation of longitudinal waves propagating through $< 110 >$ oriented single crystals of copper during cyclic deformation.

A general review "The Use of High Frequency Ultrasonic Measurements to Study the Effects of Dislocations on the Mechanical Properties of Crystalline Solids" is included as an Appendix.

The work to be accomplished during the next reporting period is included.

CONTENTS

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SECTION I

INTRODUCTION

The purpose of this research is to elucidate the basic mechanism of metal fatigue by studying dislocation dynamics in single crystals during stress cycling. The behavior of dislocations under alternating stress will be described by ultrasonic (megacyle) attenuation measurements. A basic and applied approach will be explored. The basic investigation will be limited to an extensive study of the dislocation dynamics of the first cycle of stress - a study of the Bauschinger effect. Understanding the first cycle of the fatigue process appears to be a necessary first step in arriving at an explanation of fatigue. The applied investigation will deal with an evaluation of similar measurements made continuously during cycling to fracture. Conventional S-N curves as well as attenuation versus N curves will be developed, bridging the gap between conventional S-N information and the newer type of scientific information to be generated.

The use of single crystals of definite orientation minimizes ambiguity in interpreting the damping measurements. It avoids microscopic nonuniformities associated with polycrystalline specimens and minimizes background losses due to grain boundary scattering. Furthermore, individual slip systems can be studied in single crystals because pure mode waves of a given polarization can be propagated along a definite orientation.

A large single crystal of pure copper was grown. Electrochemical cutting and chemical polishing were used to avoid introducing undesirable strains while preparing the test specimens to required dimensions. The mechanical loading system was calibrated to determine the degree of eccentricity of loading a typical test specimen in tension or compression. The reproducibility of testing was investigated. A comparison of ultrasonic attenuation between "as grown" and vacuum annealed test specimens was made. Preliminary attenuation measurements were made of the first cycle of stress of the fatigue process (Bauschinger effect).

SECTION II

ACCOMPLISHMENTS

A. GROWTH OF ORIENTED COPPER SINGLE CRYSTALS

A thirteen-inch long <110> copper single crystal was grown from a <110> seed crystal and two 4-1/2 inch lengths of 99.999^{+}% copper ingots. The crystal was grown in vacuum zone refining equipment at a rate of 0.25 inch/hr. The procedure is described in the first quarterly report.

B. ELECTROCHEMICAL CUTTING OF COPPER SINGLE CRYSTAL

The single crystal test specimens were cut to size by electrochemical cutting and chemical polishing to avoid introducing undesirable strains. Polycrystalline copper rods were used to establish processing procedures. Since the desired electrolytic action occurs at the circumference of the stainless steel discs, all other areas had to be protected with an insulative paint. Several varieties of rubber-base acid-resistant paints that were tried failed to adhere near the edges of the discs. A coating of Insulweld adhesive was effective.

Continuous unidirectional cutting produced a nonuniform cut which was wedge-shaped in cross-section. The shape was attributed to the current paths established as the electrolyte passed through the gaps between the specimen and the discs. More uniform cross-sections were obtained when the direction of the cutting discs was reversed at three minute intervals. The procedure was automated by a synchronous, reversible, D.C. motor and a timer. The smallest thickness of cut, 3/16 to 1/4 of an inch, was produced at a speed of 24 r.p.m. Removal of a slight negative potential between the specimen and the discs was necessary to prevent occasional arcing, nonuniform erosion and pitting.

High cutting speeds frequently caused arcing and short circuits when the specimen made contact with the discs. The cutting speed was controlled by a sample lowering mechanism. The optimum lowering speed was found to be 0.004 inch per minute for the current density and disc rotational speeds used. A copper specimen of 0.620 inch diameter was cut in approximately 2-1/2 hours.

After the preliminary experiments with polycrystalline copper, a number of test specimens were cut from the thirteen-inch single crystal. Laüe back reflection x-ray photographs were taken of the ends of several of these specimens. It is evident from Figure 1a., which is representative of all the specimens, that sharp clear diffraction spots are prevalent. The absence of asterism indicates that no deformations have been introduced.

C. CHEMICAL POLISHING OF TEST SPECIMENS OF COPPER SINGLE CRYSTALS

The test specimens were chemically polished to obtain necessary flatness, and parallelism of ends. Figure 2 shows the equipment used. The test specimen was clamped in its holder(*) and was moved carefully toward a cloth covered disc that revolves in an acid bath. The technique developed enabled obtaining single crystal specimens flat and parallel to within 150 millionths of an inch. The lateral movement of the specimen holder was determined by the thread of the suspension rod. A disc bonded onto the assembly was divided into divisions. The smallest division corresponded to 0.0005 of an inch of lateral movement. Smaller movements could be made by means of a Tee Handle. To avoid applying even slight pressure on the test specimen during polishing, the current of the motor was monitored. It was shown that the current measuring technique was a suitable control.

Slow speeds of 24 r.p.m. were necessary to avoid corroding the body of the test specimen. Uniform flatness of the ends were obtained by changing the polishing direction every three minutes and rotating the specimen 90 degrees in the holder. Laüe back reflection x-ray photographs of single

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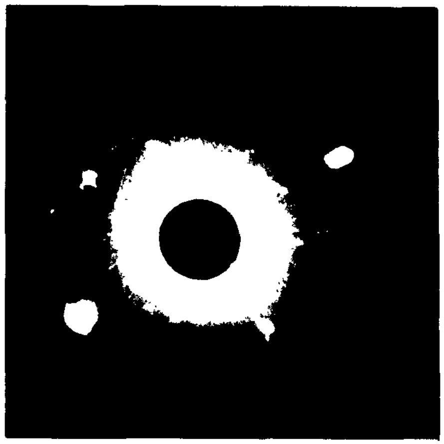
(*) The clamp was lined with foamed rubber to avoid deforming the test specimen.

a) X-Ray of Electrolytically Cut End
Specimen S1<110>3

b) X-Ray of Electrolytically Cut End
Specimen S1<110>2

c) X-Ray of End After Vacuum
Annealing at 1050°C for 137 hours
Specimen S1<110>2A



d) X-Ray of End After Chemical
Polishing
Specimen S1<110>2A

Figure 1. Laüe Back-Reflection X-Ray Photographs

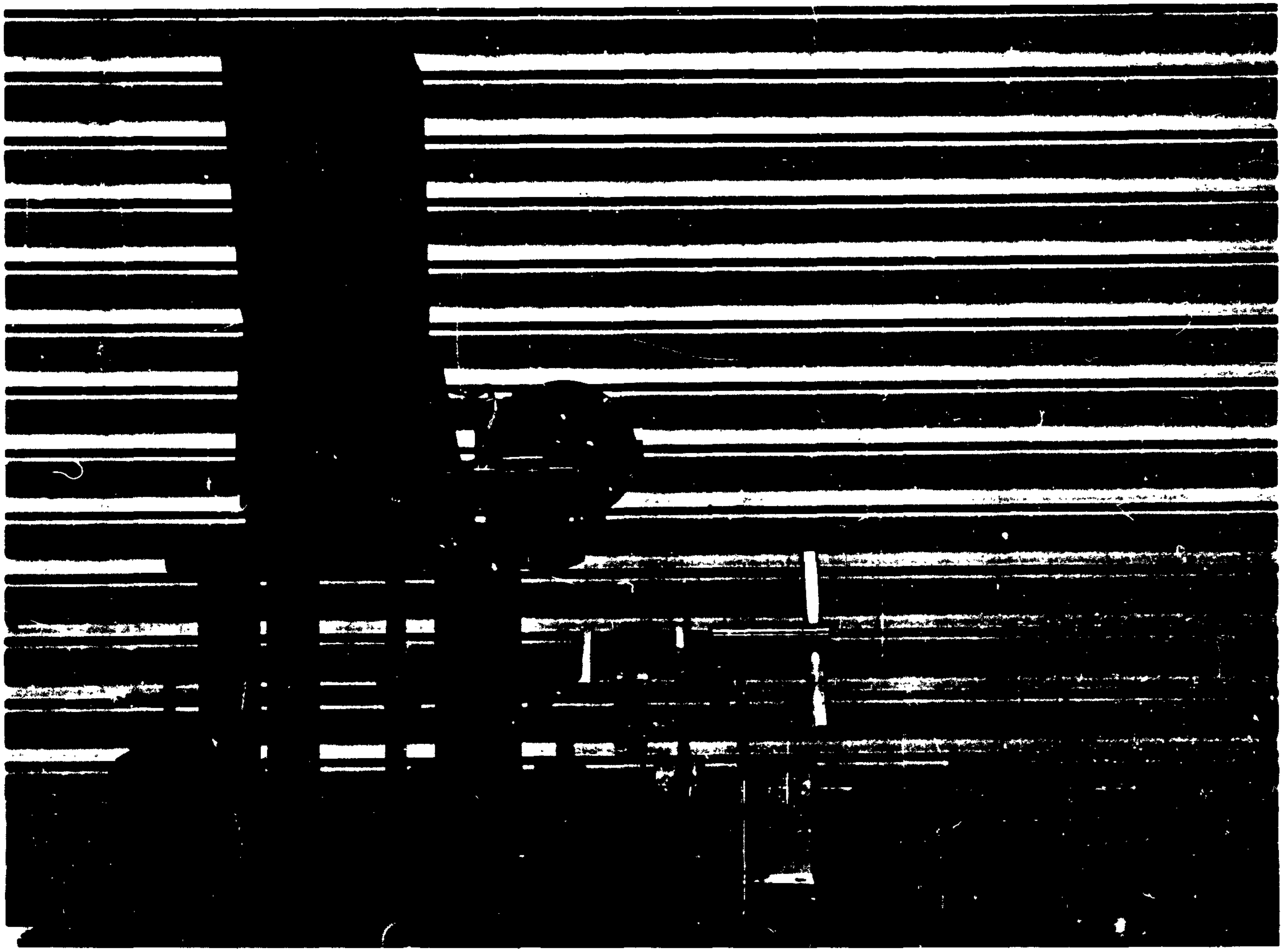


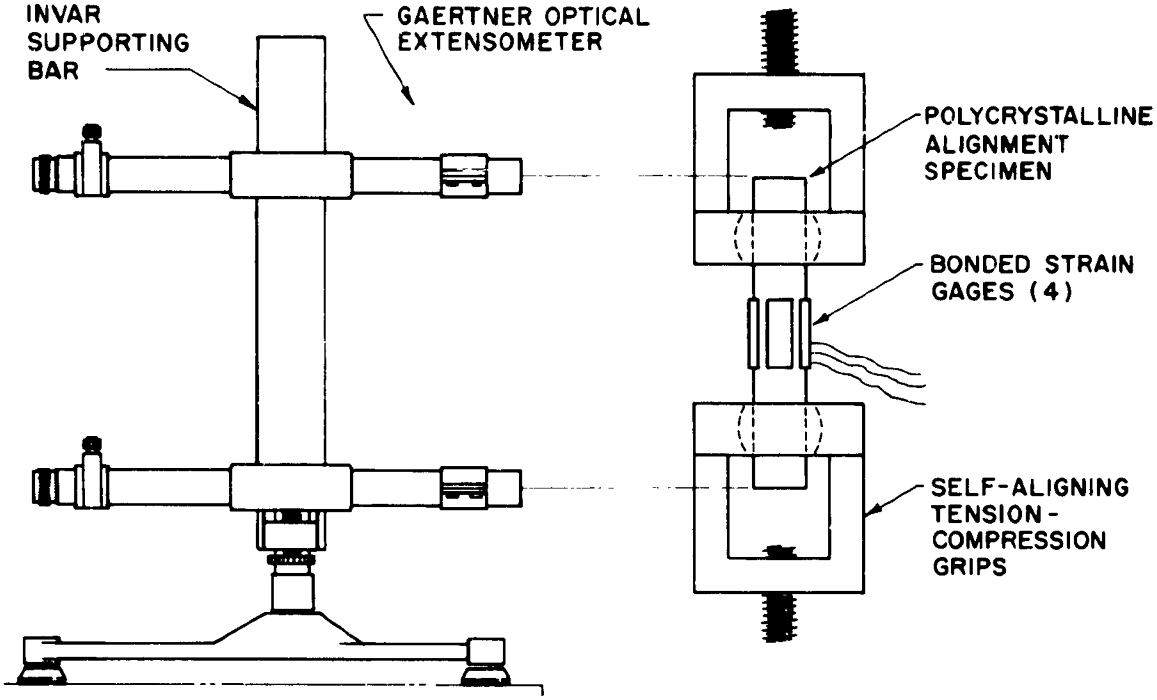
Figure 2. Chemical Polishing Apparatus

crystal Si <110> 2A showed a very slight blurring of the diffraction spots after chemical polishing. Figure 1c. is an x-ray photograph after vacuum annealing. Figure 1d. is an x-ray photograph after the same specimen was chemically polished.

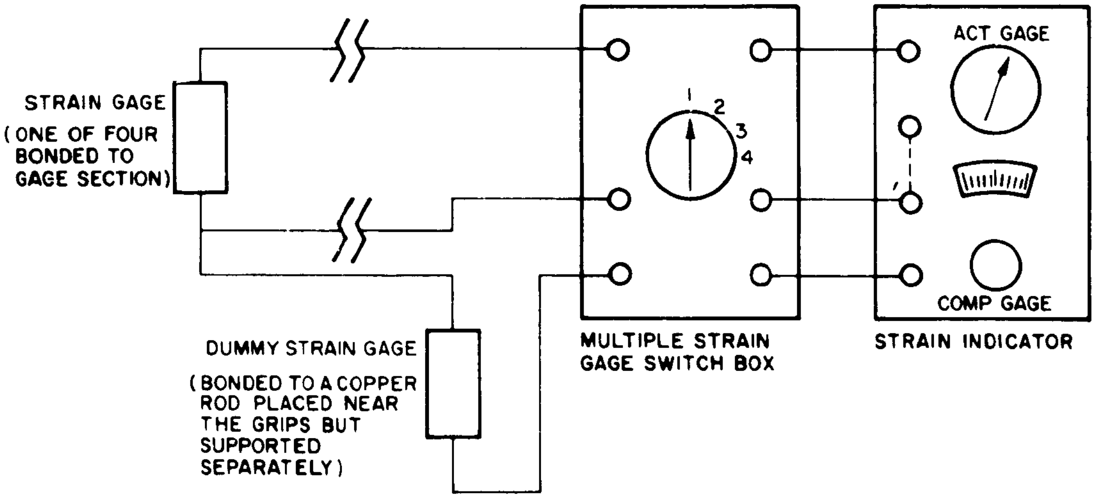
D. CALIBRATION OF MECHANICAL LOADING SYSTEM

The alignment of the extension rods of the test specimen mounting fixture was first checked by use of a Standard Pressed Steel photoelastic tensile specimen. The specimen showed no misalignment when the system was checked for tensile or compressive loads of 250 pounds. The total change in length of a polycrystalline copper test specimen was determined by a Gaertner Optical Extensometer that is sensitive to displacements of 2×10^{-7} of an inch.(*) See Figure 3a. The changes in length in a 1/2-inch gage section were simultaneously recorded from the output of four Baldwin PA5 (Post Yield) strain gages. The four gages were connected in a three wire circuit using a common dummy strain gage and a multiple strain gage switch box. The wiring circuit used is shown in Figure 3b. The output was measured in microinches by an SR4 Baldwin Lima Hamilton Strain Instrument (Model 120). Comparisons of the strain gage readings and the optical readings on two polycrystalline specimens indicate that there were no detectable changes outside the gage length for tensile and compressive loads up to 200 pounds. After many cycles of loading, the differences between the average reading of the strain gages and the optical readings in tension and compression at 200 pounds were 13.2×10^{-6} inch and 11.0×10^{-6} inch, respectively. The specific reason for the differences were not determined because they were smaller than the 2.5×10^{-5} inch per inch sensitivity of the extensometer-compressometer used in subsequent tests. The source of the differences may be attributed to bending, misalignment, reading errors, etc. The bronze balls to which the specimens are bonded are not perfectly spherical. Some misalignment may be expected because of the relatively large tolerance of 0.0015 inch - 0.002 inch that must be maintained between specimen O.D. and the ball I.D. to provide space for the bonding resin.

(*) The specimen was longer than the standard single crystal being used in order that the ends would be visible.



a) Alignment Apparatus



b) Schematic of Three Wire Strain Gage Circuit

Figure 3. Alignment Specimen and Test Apparatus

The maximum differences between the output of the four separately wired strain gages varied from 5 to 14 micro inches per 1/2 gage length.(*) These measurements were made on a second polycrystalline specimen loaded in tension over the range of 10 to 90 pounds. The differences were nonlinear with load. Retest of the specimen after 90 degrees rotation in the grips gave new differences in the range of 10 to 15 micro inches. Although it is likely that these differences may be attributed to seating irregularities and crystal misalignment, additional tests would be required for verification.

E. TESTING PROCEDURE FOR SINGLE CRYSTALS

Three single crystal test specimens were cut electrolytically from the thirteen-inch crystal. They were x-rayed to determine crystal perfection. Figure 1a. is a photograph of a typical x-ray. Specimen S1<110>2 was placed into a split graphite mold which was put in a calibrated heated vacuum retort. The specimen was annealed at 1050°C (2822°F) for 137 hours and x-rayed again. Figure 1c. shows the characteristic diffraction spots are sharp and clear. No marked differences were evident in the x-rays of the specimen after electrolytic cutting and after annealing.

The first attempt to lap a single crystal test specimen (S1<110>1) flat and parallel to 50 millionths of an inch was unsuccessful - the specimen was bent slightly. The specimen was further deformed when gage blocks used to locate the bronze gripping balls had to be removed forcibly. A technique was devised to avoid such difficulty.

The following procedure was evolved for handling the single crystal specimens after chemical polishing:

1. A 10 mc. transducer was bonded with Salol to the end of a specimen and the initial attenuation was measured.

2. The transducer was removed by immersion in hot water and the bronze balls were bonded to the specimen.

3. The bronze balls and specimen ends were lightly polished with Gamal solution, using a billiard table cloth.

(*) The sensitivity of the SR4 recorder is 5 micro inches per inch.

4. The gage section was swab-etched with dilute HNO_3.

5. The transducer was rebonded with Salol to the specimen and the attenuation was remeasured.

6. The specimen was placed in the tension-compression grips.

7. Flat positioning plates were bolted to the grips to prevent movement during transport to and installation in the test machine.

8. The grips were connected to extension rods in the test fixture and the positioning plates were removed.

9. Attenuation measurements were made at no load.

10. The compressometer-extensometer was positioned between the bronze balls.

11. Uniform seating was attained by taking up the slack at a speed of 0.0045 inch per minute.

12. The specimen was loaded in tension and attenuation readings were taken as the specimen was loaded up to 50 pounds. (See Figure 4.)

13. The upper and lower compression housings were tightened separately. The load dropped off about 15 pounds as each housing was tightened (total decrease of about 30 pounds).

14. Cyclic tests were performed at a crosshead speed of 0.0045 inch per minute.

F. PRELIMINARY ATTENUATION MEASUREMENTS

Attenuation measurements of a longitudinal wave were made on 2.6 to 2.8 inch long single crystals. Only one echo was detected in specimen S1<110>1 at 10 megacycles. This was presumably due to the deformation of the specimen in an attempt to make it flat and parallel to 50 millionths of an inch by lapping as well as deforming it during subsequent handling.

Fifteen echoes were detected in specimen S1<110>2A at 10 megacycles before the bronze balls were bonded to the test specimen. Three echoes were detected after bonding. Shifting to 5 megacycles enabled using 6 echos during the cyclic loading. Accidental preloading of the specimen while removing the slack between the balls and the tension housing reduced the echoes from 6 to 4.

Figure 4. Tension-Compression Test Fixture

Twenty-five echoes were obtained at 10 megacycles in the decay pattern for specimen S1<110>3 before the balls were bonded to the specimen. Only seven echoes were obtained after bonding.

The decrease in the number of echoes can be partially attributed to stresses introduced in the specimen during bonding the balls to the specimen. A brief investigation will be made to obtain a more compatible adhesive (i.e., lower shrinkage during cure, etc.).

Comparison of the decay patterns at 10 megacycles shows the specimens as grown attenuated less (lower internal friction) than the specimens after annealing. Their x-ray patterns show very little, if any, difference in structure. Figures 1a., 1b., and 1c. compare the as grown specimens S1<110>2 and S1<110>3 with the vacuum annealed specimen S1<110>2A. These x-rays only describe the end portions of the crystals. A more thorough x-ray analysis of the test specimen coupled with metallographic analysis would be necessary for a better understanding of these observations.

G. DISCUSSION OF INITIAL DEFORMATION STUDIES

The previous sections dealt mainly with the growth, cutting, and polishing of copper single crystals, as well as the design and calibration of the deformation apparatus. With these problems of technique resolved, reliable measurements can now be made during the cyclic deformation of oriented crystals.

The initial experiments were largely exploratory in nature and had the threefold purpose of: (1) checking the loading apparatus and extensometers during the deformation of single crystals, (2) obtaining a measure of the attenuation changes to be expected during cyclic deformation, and (3) determining the magnitudes of the loads to be used in future experiments.

The first experiment was primarily a check of the experimental setup. Specimen S1<110>1 was deformed in tension to a strain of approximately 0.1 percent. This specimen had previously been accidentally deformed and the attenuation in the specimen was very high. Only one echo was visible in the decay pattern from a 10 megacycle longitudinal wave propagating through the

specimen. The height of this echo decreased continuously during the tensile deformation. Visual measurements of the height of a single echo, however, do not give reliable attenuation measurements.

In the second experiment, both attenuation and strain were measured during two cycles of deformation of specimen S1<110>2A. At least three echoes were obtained in the decay pattern of a five megacycle longitudinal wave throughout the deformation. Reliable attenuation measurements were always possible. The results of the experiment are shown in Figure 5 in which both the load and attenuation are plotted as functions of percent strain. The specimen was first loaded in tension to point A and then cycled to an equal compressive load B and then back to the previous tensile maximum C. The tensile load was then increased to D and a new cycle was run. In this new cycle, the load was reversed, the specimen was taken into compression to a load E equal to the previous tensile maximum, and then unloaded to zero load F. The attenuation changed significantly during the tensile loadings O to A and C to D. The attenuation changes were relatively small during the cycles. This may be interpreted as signifying that appreciable dislocation locking has occurred prior to the load at point A, since four slip systems are operative. It is therefore planned to run some future experiments at lower loads. It should be noted that the attenuation due to dislocations is proportional to the resolved shear stress factor of the ultrasonic wave being propagated.(*) Using the published formulas[1] for these factors in fcc materials, one finds that the <110> longitudinal wave is less sensitive to dislocation motion than either of the two <110> shear waves. The resolved shear stress factors of the shear waves for copper should be roughly 2.5 times larger than that of the longitudinal wave. Future experiments will, of course, utilize these <110> shear waves.

In both the above experiments, Salol was used as the bonding agent between the transducer and the specimen. Other investigators[2,3] have used Nonaq grease or Eastman 910 cement, because Salol bonds have been known to "pop off" in the course of deformation. With the gripping arrangement employed in these experiments, however, it was found that the Salol bond was

(*) Cf Equation 2, Appendix

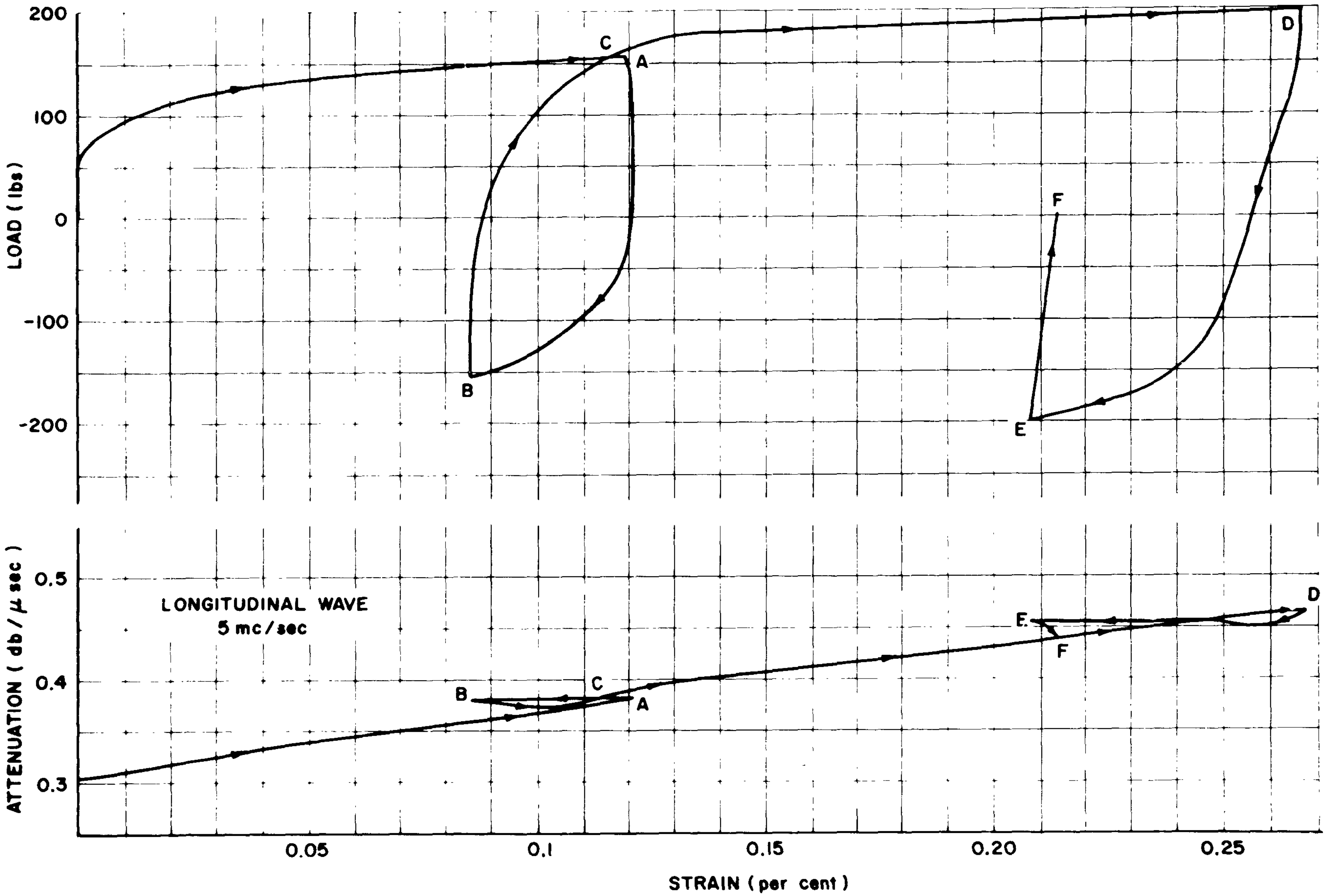


Figure 5. Load and Attenuation Versus Strain for Specimen S1<110>2A

completely reliable throughout the deformation. One of the reasons for preference of the Salol bond is that the transducer can be applied to the specimen with a minimum of pressure and handling.

A general review, "The Use of High Frequency Ultrasonic Measurements to Study the Effects of Dislocations on the Mechanical Properties of Crystalline Solids" is included as an Appendix.

SECTION III

PROJECTED WORK FOR NEXT QUARTER

The following areas will be investigated during the next quarter.

1. A brief study will be made to obtain an adhesive that will not introduce as many strains as Insulweld when used to bond the bronze balls to the single crystal test specimens.

2. Additional <110> single crystals of copper will be grown, electrolytically cut and polished.

3. Cyclic deformation studies will be performed on the <110> single crystals. The attenuation of individual longitudinal and transverse waves propagating through the specimens will be measured during the deformation. An attempt will be made to measure the attenuation of both shear and longitudinal waves of the same frequency in the same specimen during deformation. If successful, the results would be used as a direct check of dislocation damping theory (see Appendix). If the attenuation is due solely to dislocations, the ratio of the attenuation values for the two waves should be constant throughout the deformation and be equal to the ratio of the resolved shear stress factors of the two waves.

4. Single crystals of the <100> orientation will be grown and cyclic deformation studies will be performed.

APPENDIX

THE USE OF HIGH FREQUENCY ULTRASONIC MEASUREMENTS TO STUDY THE EFFECTS OF DISLOCATIONS ON THE MECHANICAL PROPERTIES OF CRYSTALLINE SOLIDS

I. INTRODUCTION

In recent years ultrasonics in the megacycle range of frequencies has become an important tool in the investigation of the nature of solids. It has been realized that the propagation behavior of high frequency stress waves in solids provides a sensitive means for the study of fundamental physical properties. The stress waves can usually be regarded as plane, and their behavior is characterized by two quantities - attenuation (a measure of the energy lost from the wave as it traverses the solid) and velocity (a measure of the dynamic modulus of the solid). Any physical process which affects the strength of a material or is capable of extracting energy from a stress wave is amenable to study by ultrasonic means. The area of solid state physics in which the ultrasonic technique has met with greatest success is that of dislocation dynamics; other areas of application include radiation damage, electron-lattice interaction, thermoelasticity, internal oxidation of metals, ferromagnetism, nuclear magnetic resonance, and the measurement of elastic constants.

The purpose of this section is two-fold: First, to explain how attenuation and velocity measurements are made in the megacycle range; secondly, after a discussion of the versatility and sensitivity of the measurements, to show how they are interpreted in terms of present theories of dislocation damping.

II. MEASUREMENT OF ATTENUATION AND VELOCITY IN THE MEGACYCLE RANGE OF FREQUENCIES

A. Technique

Equipment for measurement of attenuation by the pulse echo method is commercially available in the low megacycle range from about five to two hundred megacycles per second. Basically, the technique is as follows: A quartz transducer is bonded to a specimen on which a pair of faces have been cut plane and parallel. A signal from a pulsed transmitter operating at the fundamental frequency of the transducer or one of its odd harmonics, is applied across the faces of the transducer; through the piezoelectric effect an elastic wave is produced which propagates through the sample. The wave is almost perfectly reflected at the back air-specimen interface and returns to the transducer which converts a negligible amount of the energy of the wave back to electric energy. This electric signal is subsequently amplified and rectified by a radio frequency receiver and displayed on a cathode ray tube. In the meantime, however, the elastic wave has been reflected in the air-quartz interface of the transducer and returned to the sample. The process described above is then repeated. (The necessity for parallel faces is now apparent; parallelness is required to contain the ultrasonic beam upon each reflection within the cylindrical region beneath the transducer) Each time the elastic wave passes through the sample, part of its energy is absorbed; therefore, each signal presented to the cathode ray tube is weaker than the preceding one. The result is that there is displayed across the face of the tube a series of "echoes" ever decreasing in amplitude. Each echo is the envelope of a rectified radio frequency pulse which has passed through the sample twice since the preceding one was displayed. In order to insure that the signal is retained on the face of the tube long enough to be observed, the transmitter is pulsed at a rate which allows the entire process to be initiated once about every 10^3 echoes.

If the energy losses (attenuation) in the material are uniform without sudden large losses at any points, the decay of the sound energy will be exponential; therefore a calibrated continuously variable exponential waveform has been provided in the commercial instrument. By matching the exponential waveform to the peaks of the echoes it is possible to obtain directly the value of attenuation (the units of attenuation used are decibels per microsecond). The velocity of sound in the specimen may be readily obtained (using the same instrument) by measuring the round trip time of each echo by means of a built in time delay generator; the path length in the material divided by this round trip time will give the velocity.

B. Factors Affecting Absolute Attenuation Measurements

The accuracy of attenuation measurements is affected by many factors; we consider some of them in turn:

1. Transducer Diameter: Since the transducer is a finite source, there will be beam spread[4] - the larger the transducer, the less the diffraction. With a circular transducer, $\frac{1}{2}$" diameter, this effect is usually negligible above 50 megacycles.

2. Quality of Bond: The degree of parallelness of the top face of the transducer to the bottom face of the specimen can only be as good as the uniformity of the bond between specimen and transducer (assuming the latter are properly prepared). Since the transducer integrates mechanical energy appearing across it, it is apparent that a small degree of wedging will produce a phase shift of 180° from one edge of the transducer to the other after a few round trips in the sample. Such a phase shift may cause a complete cancellation and no output will be obtained from the transducer. After a few more round trips much of the energy may again be in phase and the echoes will increase in amplitude. A modulated envelope will be obtained.

3. Bonding Material: Since the measured attenuation is to be representative of the specimen, the energy loss in the bond must be made as small as possible. The choice of bonding material[5] depends as a rule on the type of wave to be propagated and the temperature of measurement. Salol (phenyl-salicylate - $C_{13}H_{10}O_3$) has been used by many experimenters for measurements with both longitudinal and shear waves near room temperature, the principle advantage being that a very thin seal can be obtained. Likewise Dow Corning Resin 276 can be used for both shear and longitudinal waves from -80°C to 10°C; a thin seal can be obtained by first heating to about 60°C.

For low temperature measurements the Dow Corning DC-200 silicones may be used (25°C to -270°C for longitudinal waves, -60°C to 270°C for shear waves). These fluids are also available in a wide range of viscosities, those graded 2×10^5 to 2.5×10^6 centistokes being the most useful. The higher viscosity material is particularly suited to measurements at liquid helium temperatures.

"Nonaq" stopcock grease is satisfactory for both shear and longitudinal waves over a wide temperature range: -195°C to +70°C. This material is unsatisfactory for soft specimens because of pressure required to develop good bond.

4. Specimen Preparation: Two plane, parallel faces are cut in a direction normal to that in which the wave is to be propagated. Whether the sample is machined or ground, it should be lapped afterwards to reduce surface effects on the sound wave. The lapping process should be carried to a finer final lap as the frequency of measurement is increased. (An optical polish should be obtained if possible for operation in the 100-200 mc/sec range).

If the surfaces between which the ultrasound is reflected are not parallel, part of the energy from the reflected wave does not return to the transducer, since the wave will not be reflected normal to the plane of the transducer. Also, as the reflected wave is no longer parallel to the transducer, the integrated response of the transducer is less. These effects are cumulative upon successive reflections, thus producing an apparent loss.

Another result of non-parallelism is that mode conversion will occur. Assume that a pure compressional wave is reflected from a plane boundary which is not normal to the direction of propagation. Since there is a stress component lying in the plane at the boundary, part of the energy of the compressional wave will go into shear waves, thus producing another apparent loss.

Also, if the lateral dimensions are too small compared to the path length for the ultrasound, that part of the elastic wave which does not travel normal to the transducer because of beam spread and non-parallelism of surface will eventually strike the side walls and return, after a devious route, to the transducer. These reflections are superimposed on the ordinary echo pattern and confuse the measurements.

Single crystal specimens are to be employed whenever possible, since the problems encountered with polycrystalline specimens are many. First of all, there is scattering at each grain boundary[6]; even more important there is mode conversion at each boundary. Materials showing low elastic anisotropy as a function of crystal structure orientation provide little scattering with low angle boundaries (ex. - tungsten cast by the Vernoulli technique). High angle boundaries in all materials provide appreciable sonic scattering.

The presence of anisotropy in single crystals, while of course increasing the complexity of measurement, provides a coordinate frame in which

loss mechanisms will in general imbed themselves with a preferred orientation. By probing ultrasonically in different crystalline directions with the elastic modes available, one may examine the directional properties of these loss mechanisms. Accurate phase velocity measurements when feasible are also of interest, both to correlate variations in elastic constants[7] with specimen history and to observe dispersions associated with anelastic properties. It is advantageous to choose propagation directions in the crystal for which pure transverse and longitudinal modes can be obtained.[8]

Using X-cut and AC-cut quartz transducers, one may excite various modes individually instead of in proportion depending on the orientation of the propagation direction. The computation of elastic constants from measured phase velocities is then less involved. Further, the analysis required to explain observed losses is usually simplified because the crystal and hence many of the loss mechanisms, will have a fairly high symmetry in the propagation coordinates. Again, the direction of energy flux will almost always coincide with the propagation direction, so that pulses reflecting between two parallel crystal faces will not deteriorate due to impinging on side walls of the crystal. (In actual practice it is impossible to cut these faces to exactly the desired orientation.)

Presumably extremely careful machining techniques can hold misorientation to a matter of several minutes of arc. In easily damaged specimens, however, which cannot be machined, misorientations incurred in a lapping procedure may easily be of the order of one or two degrees. There are then energy flux deviations which influence loss measurements.

All of the aforementioned factors influence the measurements to such a high degree that absolute attenuation measurements are very difficult to make. Therefore, most work is done on a comparative basis where changes in attenuation are measured as a function of other parameters such as strain, mag-

netic field, temperature, etc. (If measurements need be made in various samples as a function of say chemical composition, it is essential that the samples have the same geometry, that the transducers be identical, and that great care be taken in making the bonds to insure that they are uniform and that the samples be identical in all respects except for the one parameter that is under investigation).

III. SELECTIVE APPLICATIONS OF HIGH FREQUENCY DAMPING MEASUREMENTS

Ultrasonic measurements has found its greatest application in the fundamental study of the mechanical properties of crystalline materials. There are interactions between dislocations and ultrasonic waves,[9] interactions which permit the study of dislocation dynamics, particularly as the dynamics are influenced by interaction with point defects. The methods are particularly sensitive to changes in the nature and number of defects present as well as to the manner in which the defects are distributed in the material. Damping measurements have been used to follow the behavior of dislocations and point defects during plastic deformation in metals[10], recovery of mechanical properties[11], neutron and gamma irradiation of metals[12] and semiconductors[13], and diffusion of impurities[14]. It should be pointed out that in the study of imperfections ultrasonic techniques are usually more sensitive than other types of measurement. For example, in stressing a solid at relatively low stresses the stress-strain law may appear linear but of course always contains an anelastic component, no matter how small. Damping measurements "see" only this anelastic component; the elastic strain, since it is reversible, does not contribute to the damping. As another example, consider a recovery process where point defects migrate to dislocations. The damping is influenced by the very beginning of the recovery process, i.e., by the first of the defects to migrate. This occurs since the damping depends very sensitively on the length of vibrating dislocation (i.e. the average distance between pinning points) and the first few pinning points may be sufficient

to reduce the damping to negligible values. On the other hand, resistivity measurements (for example) depend on the number of defects which remain in the lattice. This number will not change appreciable until nearly the end of the migration process; thus the corresponding resistivity changes would occur at later times.

The interpretation of attenuation and velocity measurements requires a quantitative theory of dislocation damping. Recently, several theoretical models, which seem to be capable of describing at least certain aspects of the damping observed, have been proposed. These are discussed in the next section.

IV. DISLOCATION DAMPING

A. Mechanisms Involving Dislocations

In 1940, Read[15] in a series of internal friction experiments on slightly deformed single crystals of copper and zinc was able to demonstrate convincingly that dislocations give rise to mechanical energy losses in crystals. Although this work and similar investigations on the dependence on crystal orientation and on the recovery of stress-induced internal friction of crystals had very early established the importance of dislocations (see Seitz's book[16], 1943), it was not until recently that a detailed picture of at least some aspects of the dissipating mechanism due to dislocations began to emerge. In Zener's book[17] (1948) on anelasticity of metals dislocations are barely mentioned as contributing to energy dissipation, and even in 1952 Wert[18] stated that the internal friction measurements on plastically deformed crystals have failed to tell us how the dislocations involved were arranged and anchored, and with what types of dislocations we are dealing.

Two main reasons are held responsible for the slowness of progress in the field. First, the internal friction of single crystals is sensitive to static stresses even below the critical shear stress τ_o. As reported by most experimental investigators internal friction measurements on metal single crystals

are therefore extremely sensitive to handling damage. Secondly, the internal friction depends on the magnitude of the amplitude of the alternating strain employed in the measurement well below the critical shear strain ε_o (in metal crystals, $\varepsilon_o \simeq 10^{-5} - 10^{-4}$). Since the theory of dislocations was developed mainly to account for the plastic properties on a stress level $\tau \geq \tau_o$ the internal friction experiments involved dislocation properties on which no detailed theories were available. On the other hand, the damping experiments provide an excellent method of studying dislocations on a stress level to which there is no easy access.

The mechanical energy losses caused by the movement of dislocation which have been discussed in the literature can be grouped in three classifications:[19] hysteresis losses,[20,21] resonance losses,[22] and relaxation losses[23].

The great majority of the energy losses by dislocation processes is of the hysteresis type. Typical hysteresis processes are e.g. those in which dislocations are torn away irreversibly from pinning points by the applied stress. Such pinning points are provided by foreign atoms or other point defects defects[12, 22, 24, 25, 26] and by other dislocation lines intersecting the area swept out by the moving dislocations.[27]

A dislocation line fixed at two points behaves somewhat like a string. It can undergo resonance if the frequency f of an applied alternating shear stress coincides with the characteristic frequency f_o of the dislocation string. Plotted as a function of frequency the internal friction should go through a maximum at f_o (or near to the maximum of a distribution of resonance frequencies).

A typical relaxation process in crystalline solids is the Snoek[28] process of carbon atoms in α iron. Its characteristic features are as follows: in thermal equilibrium the physical system under consideration passes with a temperature-dependent mean frequency ν from one configuration of minimum

energy through a saddle point configuration to one of several other minimum configurations. An applied stress favors in general some of these minimum positions with respect to others. If we apply an alternating stress of frequency f, well known arguments show that the energy dissipation is a maximum if the two frequencies f and ν are equal.

One process in crystals which involves dislocations only and which shows all characteristic features of a relaxation mechanism is that discovered in various f.c.c. metals by Bordoni[23]. The relaxation peak can be explained by assuming that dislocations lying nearly parallel to the Peierls* valleys throw loops over the Peierls hills into the next valley under the influence of the external stress with the help of thermal fluctuations.

B. Theories of Dislocation Damping

All models proposed so far attribute the dislocation damping to the vibration of dislocations, caused by the cyclic external stress.** If the dislocations could move freely, these vibrations would lead, because of the fact that dislocation motion is damped, to energy losses much larger than those observed. The dislocation motion must therefore be restricted, and the various theories of damping are differentiated only by different assumptions about the nature of the restructions, and eventually about the mechanism of overcoming them. The restrictions which have been proposed (besides the nodes of the dislocation network) are:

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* In a dislocation which lies parallel to a direction of close packing in its slip plane-for instance, the [110] direction in the (111) plane in a face-centered cubic metal-there will be at each position of the dislocation in the plane a given pattern in which the atoms are arranged with respect to the center of the dislocation. As the dislocation moves this pattern changes continuously; but because of the periodicity of the lattice, the pattern is repeated each time the dislocation moves a distance equal to the separation between close-packed rows. Thus the energy of the dislocation oscillates periodically as the dislocation moves through the lattice. This variation can be represented by a periodic potential, called the Peierls potential.

** A comprehensive review on dislocation damping in metals has been given by Niblett and Wilks.[29]

(1) Peierls hills: This restriction is used to explain Bordoni peak i.e. a maximum in the internal friction measured at a fixed frequency as a function of temperature. As stated above it is assumed that dislocations lying nearly parallel to the Peierls valleys throw loops over the Peierls hills into the next valley under the influence of the external stress with the help of thermal fluctuations. In materials studied thus far the peak temperature has varied from 36°K (in Pb)[30] at a frequency of 10 kilocycles to 180°K (in Al)[31] at 60 megacycles. A theory for this energy loss has been given by Seegar, Donth and Pfaff.[32] We should note that this theory makes use only of the small fraction of dislocations which lies nearly parallel to the Peierls valleys.*

(2) Other dislocations: The interaction of dislocations with each other will play an important role in damping in heavily deformed materials. It is possible that dislocations will be close enough together that they are immobilized. There is at present no satisfying quantitative theory of damping in heavily deformed material, since the theory of Weertman and Koehler,[34] the only one published so far, is based on a questionable arrangement of dislocations.

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* Another mechanism has been proposed by Bruner[33] for dislocation relaxation in which the essential feature is the thermally activated motion of paired partial dislocations between vacancy pinning points. This mechanism predicts the absence of Bordoni peaks in bcc pure metals. Although Bruner's measurements on zone refined iron lend substantial support to his theory, there is a need to study bcc metals of lower total interstitial content.

(3) Point defects: There are a number of ways point defects (usually foreign atoms) can restrict dislocation motion. One possibility is that the point defects follow the vibrating dislocations by diffusion causing a phase lag between the stress and dislocation motion and hence a damping. This can happen, however, only at low frequencies or at high temperatures. No thorough investigation of this possibility has been made.

A second mechanism derives from the fact that foreign atoms in solid solution cause a stress field with wavelength $\lambda = a/c^{1/3}$ where "a" is the atomic spacing and "c" is the concentration of foreign atoms. Weertman and Salkovitz[24] have developed a theory which describes the motion of dislocations (non-pinned) through this stress field. A comparison of this theory with experimental data leads to numerous difficulties, however, and it is concluded that the theory cannot have general validity.

Lastly, a theory originally proposed by Koehler[22] and then developed by Granato & Lucke[25] assumes that dislocations are pinned by foreign atoms. The loops between pinning points vibrate under the influence of the external stress and the energy loss results from damping of the dislocation motion; at large strain amplitudes the dislocation can break away from the impurity atoms leading to a hysteresis loss. In other words the model leads to two different types of loss; a frequency dependent, strain amplitude independent loss which is important in the megacycle range of frequencies, and a strain amplitude-dependent loss which is of importance in the kilocycle range of

frequencies. (In the original work of Granato-Lucke, thermal considerations were neglected so that this second loss was purely a static hysteresis. The G-L theory is being extended and modified to include thermal effects; it turns out that the strain-amplitude dependent loss is also relaxational in character) We should point out that the G-L theory has met with considerable success in the interpretation of numerous sets of experimental data.[36]

V. INTERPRETATION OF ATTENUATION AND VELOCITY MEASUREMENTS IN THE MEGACYCLE

A. RANGE

We come now to the practical application of the information contained in the above sections. First of all, we consider temperatures well above that at which the Bordoni peak occurs. With the commercially available attenuation unit the strain amplitudes are less than 10^{-7}. Hence the only damping mechanism with which we need be concerned is given by the dynamic loss* in the Granato-Lucke theory.

The mathematical model of a pinned-down dislocation loop under the influence of an applied stress is taken to be

$$A\frac{\partial^2 \xi}{\partial t^2} + B\frac{\partial \xi}{\partial t} - C\frac{\partial^2 \xi}{\partial y^2} = \sigma b \tag{1}$$

i.e. the equation for forced vibrations of a damped string. ξ is the displacement of the dislocation, b is the Burgersvector, A the effective mass per unit length ($A \simeq \pi \rho b^2$ where ρ is the density of the material), the term in B is the damping force per unit length, and C is the effective line tension[38] ($C \simeq Gb^2$ where G is the shear modulus).

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* Recall that we are mainly interested in relative attenuation values. We have seen in a previous section how to minimize background and geometrical losses. Scattering losses will be unimportant in a relatively pure single crystal; in a polycrystal the grain boundary scattering loss will be constant during any physical process which would be under investigation e.g. irradiation, composition studies, recover, etc. In any event, scattering losses can be checked since they have a unique frequency dependence.[37]

Contributions to the expected damping constant B have been calculated by three authors. Eshelby[39] considers the loss due to the irreversible heat flow which occurs in the neighborhood of an oscillating dislocation; Leibfried[40] estimates the loss associated with the interaction of a moving dislocation with thermally excited sound waves; Mason[41] discusses the effect of phonon viscosity on dislocation motion. In any event there is a loss associated with this model due to the fact that the motion of the dislocation is opposed by some damping mechanism, so that there is a phase lag between its displacement and the applied stress. This loss is frequency dependent, since it has a resonance-type character. For low frequencies, the motion can follow the applied stress and the decrement is small. Near the resonant frequency, determined by the loop length, the motion is out of phase with the applied stress by 90° and the decrement* is a maximum. The logarithmic decrement Δ and the modulus change $\Delta E/E$ are given according to Granato and Lucke by

$$\Delta = \pi \Omega \Lambda \left(\frac{C}{A}\right) \frac{\omega d}{(\omega_0^2 - \omega^2)^2 + (\omega d)^2} \tag{2}$$

$$\frac{\Delta E}{E} = \Omega \Lambda \left(\frac{c}{a}\right) \frac{(\omega_0^2 - \omega^2)^2}{(\omega_0^2 - \omega^2)^2 + (\omega d)^2} \tag{3}$$

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* The logarithmic decrement Δ for any wave is given (for sufficiently small α) by $\Delta = \alpha \lambda$, where λ is the wavelength and α is given in nepers per unit length. Values for attenuation $\alpha' = \alpha v$ used in the literature are given in decibels mer microsecond. This corresponds to $10^6/8.68$ nepers per second.

where:

Ω is an orientation factor taking into account the fact that the resolved shear stress on the slip planes is less than the applied stress

Λ is the dislocation density

ω is the frequency

$\omega_o = \pi C^{1/2} A^{-1/2} L^{-1} t_1^{-1/4}$, the resonant frequency of the dislocation

t_1 is a numerical factor depending on the distribution of loop lengths

L is the average loop length and d = B/A

For large damping the decrement has a maximum at w_o^2/d. (Using estimated values of the constants it is found that a maximum damping of this type would be expected in the megacycle range).

For frequencies much less than that for which the decrement is a maximum, equations (2) and (3) become*

$$\Delta = \Omega \Lambda L^4 B \omega t_1 / \pi^3 C \tag{4}$$

$$\frac{\Delta E}{E} = \Omega \Lambda L^2 t_2 / \pi^2 \tag{5}$$

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* t_1 and t_2 are factors which depend on the distribution of loop lengths assumed. For a delta function distribution t_1 and t_2 are both unity. For an exponential distribution of loop lengths, which is obtained if the pinning points are distributed randomly over the dislocation line, t_1 and t_2 take on the values 120 and 6 respectively.

The Granato-Lucke theory is the only theory at present which predicts that the decrement and modulus change should depend on the fourth and second power of the loop length respectively. It is difficult to determine these laws if measurements on different speciments are compared. This is because the dislocation density is unlikely to be exactly the same for two different specimens, especially if the impurity content differs. What is needed is an experiment for which the loop length is changes, but not the dislocation density.

Thompson and Holmes[12] have made measurements on high purity copper during neutron irradiation. It is reasonable to assume that the dislocation density remained constant during the experiments so that the changes ovserved are all due to the changing loop length, which decreases as a result pinning by the point defects created by the irradiation. Under these conditions Thompson and Holmes show that the decrement and modulus changes are proportional to L^4 and L^2 respectively.

Recovery measurements can be used under the assumption that the recovery of damping and modulus changes is not due to annihilation or rearrangement of dislocations, but rather to the migration of point defects to the dislocations. On this basis Granato and Lucke[11] analyzed measurements by Gordon and Nowick[42] of the recovery of NaCl after deformation. Besides confirmation of the loop-length dependence they found that the concentration of pinning points followed the strain-aging law of Cottrell and Bilby.[43]

On the basis of this theory, recovery measurements in aluminum and copper also have been analyzed by Hikata and Granato[11], who find evidence for the L^4 and L^2 dependence in 14 different cases.

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